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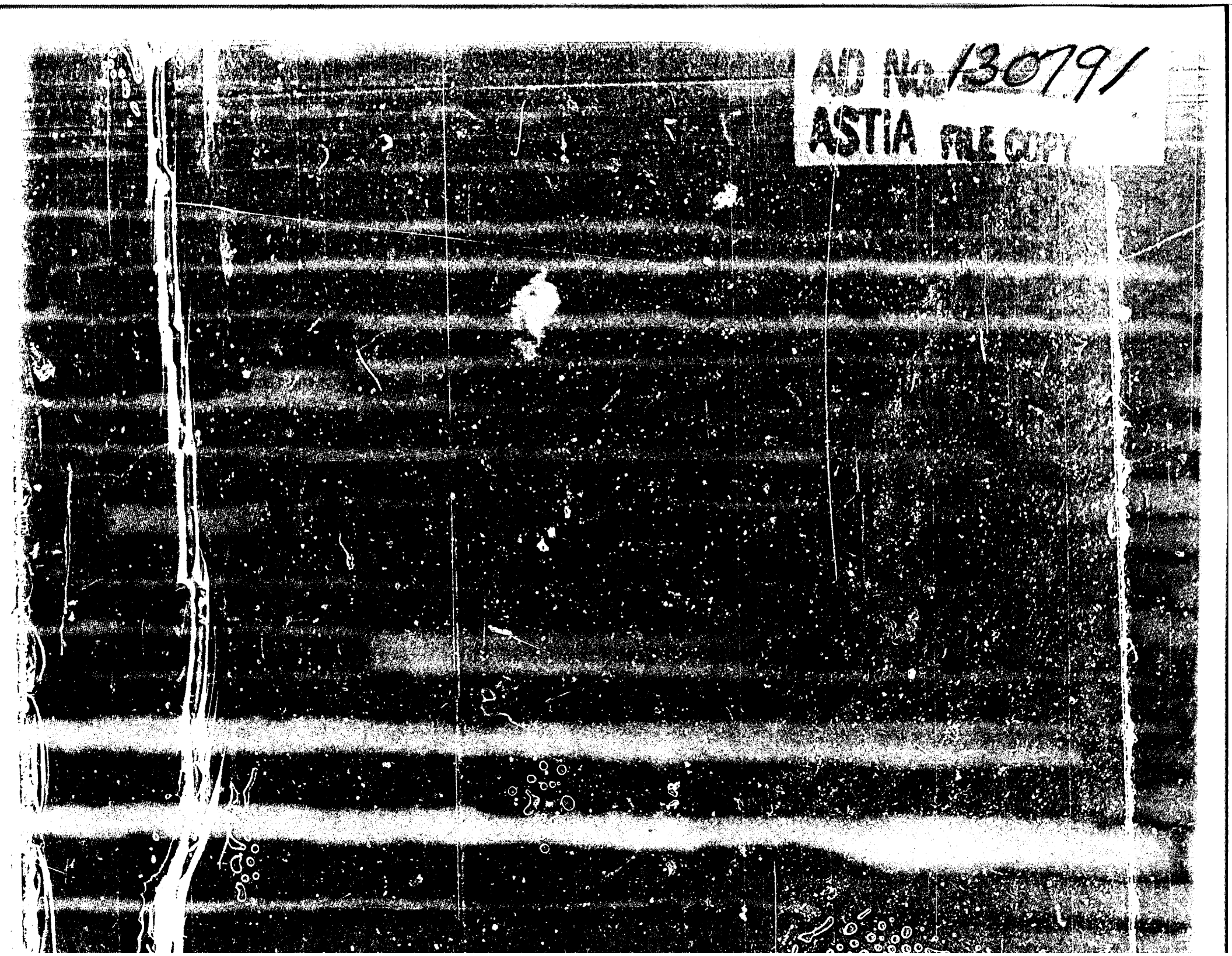

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FOR[illegible] TITLE

[illegible] EVALUATION OF [illegible] CERAMIC AND CERMET MATERIALS
FOR BEARING AND SEAL APPLICATIONS [illegible]
POWER UNITS AND [illegible] SYSTEMS

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Wright-Patterson Air Force Base, Ohio

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FOREWORD

This report was prepared by Battelle Memorial Institute under USAF Contract No. AF 33(600)-28610. This contract was initiated under Project 7350, "Ceramic and Cermet Materials," Task No. 73500, "Ceramic and Cermet Materials Development". The work was administered under the direction of the Directorate of Development and the Directorate of Research, Wright Air Development Center, with Lt. J.C. Tinius of Power Plant Laboratory and Capt. I.K. Holdener and Lt. W.P. Heuli of Materials Laboratory acting as project engineers.

This report covers work conducted from January 12, 1956 through November 11, 1956, inclusive.

[redacted] document, including the title, is classified Confidential in its entirety because of the nature and [redacted] of the research work and data described herein.

ABSTRACT

A preliminary investigation of the high-speed-rubbing wear characteristics in JP-4 jet fuel, in JP-X rocket fuel, and in hot oxidizing gas (essentially air, up to 1400 F) and the static corrosion resistance in inhibited red fuming nitric acid of selected materials has been conducted. The apparatus and the evaluating procedure for simulating the wear conditions of face seals [illegible] specimens has been developed. Experimental results with alumina, boron nitride, mica, and other ceramics and metals are included. Some tentative correlations of the results with field [illegible] presented.

[illegible] REVIEW

[illegible] and is approved.

FOR THE COMMANDER:

Richard H. Kennedy

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MATERIALS IDENTIFICATION CODE

for WADC, TR 57-4__, Table I.

Material	Supplier	Designation
Alumina, high density	Norton Company	Alundum, hot pressed
Alumina-chromium	Haynes-Stellite Company	Metal-cera... LT-1
Alumina Coating A	Norton Company	Rokide "A"
Alumina Coating B	Linde Air Products Company	Flame coat... LA-2
Alumina porcelain	Coors Porcelain Company	Type AB-2
Boron carbide	Norton Company	Norbide
Boron nitride	National Carbon Company	Type GCH
Graphite A	Stackpole Carbon Company	Grade 304
Graphite B	Stackpole Carbon Company	Grade 469
Chromium carbide-nickel	Firth Sterling, Inc.	Grade CR-2
Cobalt-base Alloy A	Haynes Stellite Company	Alloy No. 3
Cobalt-base Alloy B	Haynes Stellite Company	Alloy No. 6
Iron-base Alloy C	Haynes Stellite Company	Alloy No. 93
Nickel-boron nitride-mica	Brush Beryllium Company	Lot HP-157A-1A
Nickel-mica	Brush Beryllium Company	Lot HP-113
Stainless steel	Carpenter Steel Company	AISI 440 C
Synthetic mica	Brush Beryllium Company	Lot HP-235-18-AN
WC-TaC-Co	General Electric Company	Carboloy 907
WC-platinum	Kennametal, Inc.	K-501
Zirconia coating	Norton Company	Rokide "Z"

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A LABORATORY EVALUATION OF SOME CERAMIC AND CERMET MATERIALS FOR BEARING AND SEAL APPLICATIONS IN AIRCRAFT AUXILIARY POWER UNITS AND LIQUID ROCKET MOTORS

INTRODUCTION

In recent years the demand for higher flight speeds and greater performance of military aircraft has forced the operating conditions of many engine components beyond the capacity of conventional materials. The recent technological advances in high-temperature engineering materials has been tremendous. However, as the temperature and corrosiveness of the environments in modern aircraft power plants has increased, the materials used for rubbing wear components in seals and bearings have become particularly critical. Efforts to cool these parts and to isolate them from corrosive fluids have merely served to reduce the efficiency of mechanisms below that potential made possible through the use of modern, high temperature, structural alloys.

In order to assess the present state of material development for aircraft rubbing wear applications, a survey of 34 users and manufacturers of bearings, seals, ceramic materials, accessory equipment, and aircraft engines was conducted (Battelle Special Report No. 122, dated October 31, 1956). It was found that, although some independent research programs are being conducted on gas turbine power plant main shaft bearings and seals, the research effort for accessory equipment and rocket applications is restricted to the equipment manufacturers who, quite naturally, are concerned with the solution of their immediate bearing and seal problems. These groups are the manufacturers of aircraft auxiliary power units (APU), aircraft accessories, and rocket motor liquid propellant pumps. The environmental conditions for bearing and seal operation in these applications are varied and strenuous. These manufacturers require assistance in the development of materials and designs for their bearing and seal applications if the increased performance of these mechanisms is to be realized in the near future.

This research program was initiated on the current contract to provide a preliminary study of a few commercially available ceramic and cermet materials in some of the environments that are encountered in bearings and seals in APU and rocket applications. The effort devoted was intended as the beginning of a comprehensive program of materials development and evaluation to fill this void in the technological advancement of aircraft power plant systems.

EXPERIMENTAL PROCEDURES AND RESULTS

A selected number of materials have been evaluated for high-speed rubbing wear and corrosion resistance in four different environments, chosen to represent a cross section of the conditions encountered in the vicinity of the bearings and seals in current and anticipated APU and liquid rocket propellant pumps. These environments were as follows:

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(1) 200°F, JP-4 jet fuel

(2) 80°F, JP-X rocket fuel

(3) Oxidizing gas (essentially air), up to 1400°F

(4) 160°F, inhibited red fuming nitric acid (IRFNA).

High-speed rubbing wear evaluations were conducted in the first three of these environments, while the experiments in the nitric acid were limited to the static corrosion of selected materials.

The temperatures of the liquid fuel environments were the maximum that could be maintained in the apparatus without excessive evaporation.

Rubbing Wear Evaluations

Description of Apparatus Used in Rubbing Wear Evaluations

The rubbing wear evaluations were conducted on a piece of available apparatus that had been modified for these experiments. A photograph of the equipment is shown in Figure 1. This apparatus featured a high-speed spindle and timing belt drive capable of spindle speeds up to 16,000 rpm. An adapter was constructed so that a four-inch-diameter, washer-shaped specimen of a prospective seal material could be mounted on the end of the spindle and held to less than 0.0003-inch total indicator reading run-out of the front face. Three, one-half-inch-diameter button specimens of the mating seal material were mounted in a holder and lapped so that their faces were all in the same plane within 0.00001-inch deviation across their contacting surfaces. The button specimens in their holder were mounted on the end of another, stationary spindle, through a self-aligning, ball and socket joint. The lapped faces of the button specimens pressed against the front face of the rotating washer-shaped specimen. Thus, the contacting surfaces were held parallel to each other to minimize any fluid wedge action at these surfaces. The resulting fluid film thickness should be in the same order of magnitude as that found in face-type shaft seals. The rubbing surface speed between the specimens was about 200 feet per second, which is representative of the surface speeds in most APU and rocket propellant pump face seal and sleeve bearing applications.

This method of material evaluation was chosen to simulate as closely as possible the type of rubbing wear that is encountered in face seals and, at the same time, to utilize small specimens of simple shape that can be fabricated easily from a variety of materials. Of course, a more exact evaluation of seal materials for a particular application would be accomplished if actual seal components were fabricated from the prospective materials and were evaluated under service conditions. However, by keeping the specimens simple in shape, the evaluation of materials that would be

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1. Washer specimen
2. Button specimen holder and specimens
3. Rotating spindle
4. Stationary spindle
5. Test chamber
6. Test-chamber lid
7. Gas burner
8. Exhaust pipe
9. Ball-bushing, linear-motion spindle guide
10. Restraining arm for measuring friction torque

FIGURE 1. EQUIPMENT FOR THE RUBBING WEAR EVALUATION OF PROSPECTIVE SEAL AND BEARING MATERIALS IN A HOT OXIDIZING GASEOUS ENVIRONMENT

difficult to fabricate into seal rings can be accomplished much more quickly and economically. The equipment used in this program was designed to screen materials, both commercial and experimental, for their rubbing wear characteristics under conditions simulating the field service in face seals.

The stationary spindle, on which the button specimens were held, was mounted on a steel base plate. It was restrained from rotating by a cantilever member on which strain gages were mounted for recording the friction torque at the rubbing surfaces of the specimens. The rotating spindle was mounted on linear-motion ball-bushings in such a way that it was free to move axially with respect to the stationary spindle. The load on the specimen rubbing surfaces was controlled by varying the air pressure in a metallic bellows that was attached to the base plate and was pressed against the rotating spindle housing.

The ends of the two spindles, together with the specimens, were enclosed in a test chamber into which these environmental fluids were introduced. The two liquids, JP-X and JP-4, were supplied in a jet on the inner track of the rotating specimen at a flow rate of about 0.8 pounds per minute. This amount was sufficient to bathe the contacting surfaces with the liquid and to maintain a heavy mist in the test chamber. The hot gaseous environment was supplied by a gas burner similar to those in the combustion section of an aircraft gas-turbine power plant. A cross section of this burner is shown in Figure 2. The hot combustion products of natural gas and air were mixed with additional air in this burner and were introduced into the test chamber through the inlet shown in Figure 1. The gaseous environment that was supplied in this manner simulated the atmosphere in the turbine section of a gas turbine engine. The slight reduction in the oxygen content from that of normal air did not effect appreciably the oxidizing characteristics of this environment.

Description of Test Procedures in Rubbing Wear Evaluations

The specimens of two prospective seal materials were prepared by grinding, lapping, and lead lap polishing the rubbing contact surfaces to the best finish that could easily be obtained and to a flatness of less than 0.00001-inch deviation. The composition and properties of the two materials that were studied in this program are listed in Table 1. After assembling the two materials for each wear evaluation in the equipment, the specimens were brought into contact under a nominal load pressure of 5 psi on the contact area; the environmental fluids were introduced and the rotating specimen was brought up to speed. The friction at the rubbing surfaces was monitored carefully as the load was increased in 5-psi increments to 20-psi contact pressure. After an initial increase in friction following an increase in load, the friction usually settled down to a steady-state value that was somewhat higher than the friction for the lower load level. The load was not increased again until this steady-state value had been reached. The evaluations in the hot-air environment were started at about 400 F air temperature and 5-psi contact pressure. The air temperature was increased gradually up to 1400 F before an attempt was made to increase the load pressure. The specimens were operated for one hour at 20-psi contact pressure in all evaluations unless a failure caused premature shutdown of the equipment. Failures were usually indicated by an abrupt increase in friction and noise.

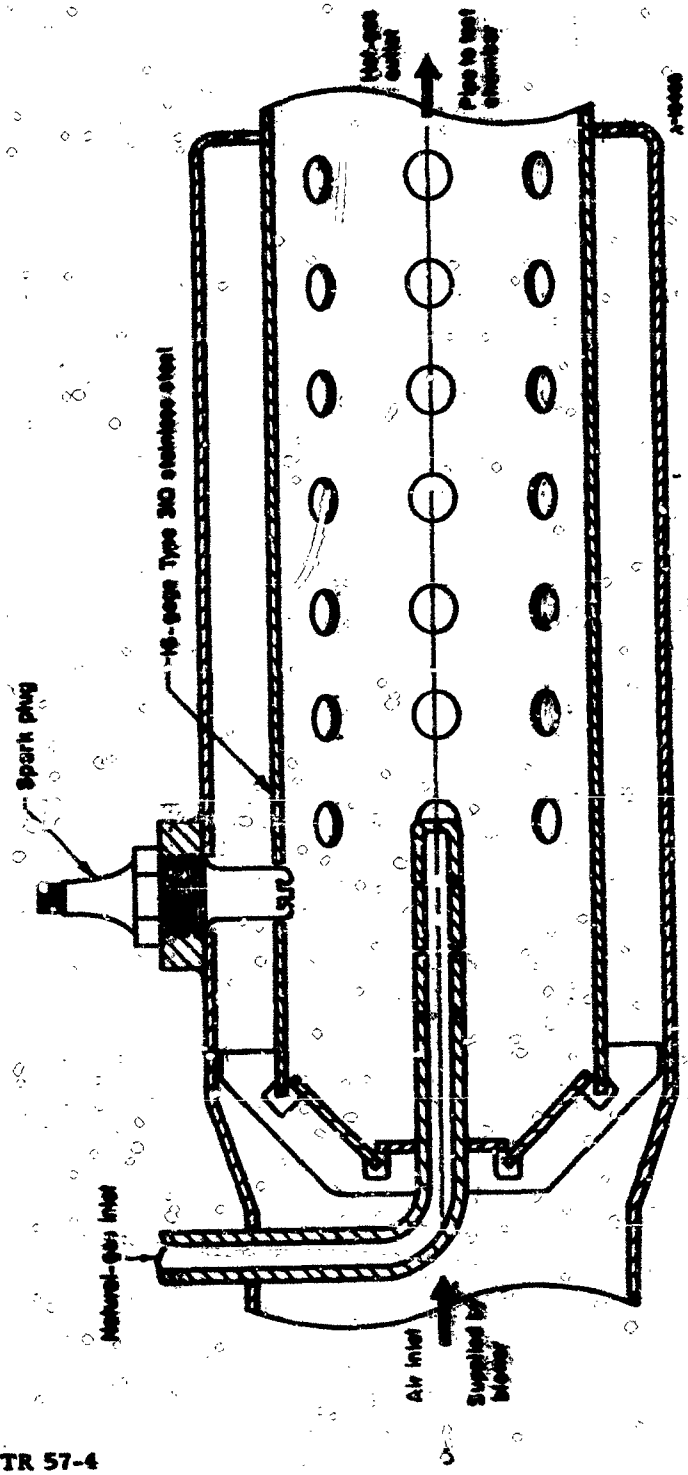

FIGURE 2. CROSS SECTION OF THE GAS BURNER FOR PRODUCING A HOT OXIDIZING GASEOUS ENVIRONMENT

TABLE 1. KNOWN PROPERTIES OF PROSPECTIVE SEAL AND BEARING MATERIALS

(Source: Trade Literature)

Material	Major Constituents in Composition, by weight	Strength, psi	Hardness	Comments
Alumina, high density	98.5% Al_2O_3	40,000 transverse rupture	2000 Knoop (K100)	Hot pressed
Alumina-chromium	77% Cr, 23% Al_2O_3	38,000 tensile	37 R_C	
Alumina Casting A	98.6% Al_2O_3	--	2000 Knoop	10% porosity
Alumina Casting B	99+% Al_2O_3	--	1000-1200 VHN	Less than 1% porosity
Alumina porcelain	86% Al_2O_3	17-18,000 psi tensile	1450 Knoop	
Boron carbide	pure B_4C	35,000 transverse rupture	2800 Knoop (K100)	
Boron nitride	BN, bonded with another boron compound	--	--	
Graphite A	Carbon	10,000 transverse rupture	50 Scleroscope	Impregnated for strength
Graphite B	Carbon	8,000 transverse rupture	75 Scleroscope	Oxidation resistant, electrographitic grade
Chromium carbide-nickel	86% chromium carbides, 14% Ni	120,000 transverse rupture	86.5 R_A	
Cobalt-base Alloy A	46% Co, 31% Cr, 12.5% W, 2.4% C	60-65,000 tensile	51-54 R_C	High-temperature wear-resistant alloy
Cobalt-base Alloy B	56% Co, 30% Cr, 8.5% W, 1.1% C	110-120,000 tensile	39-43 R_C	High-temperature wear-resistant alloy
Iron-base Alloy C	53% Fe, 17% Cr, 16% Mo, 6.25% Co, 1% C	90,000 tensile	60-62 R_C	High-temperature wear-resistant alloy
Nickel-boron nitride-mica	--	--	--	
Nickel-mica	--	--	--	
Stainless steel, hardened	17% Cr, 0.75% Mo, 1% C, bal Fe	285,000 tensile	56 R_C (455 Brinell, annealed)	AISI 440 C
Synthetic mica	--	--	--	
WC-TaC-Co	74% WC, 20% TaC, 6% Co	315,000 transverse rupture	91.3 R_A	
WC-platinum	WC, infiltrated with platinum	--	--	
Zirconia coating	98% ZrO_2		750 Knoop	

Results of Rubbing Wear Evaluations

A summary of the high-speed-rubbing wear evaluations is presented in Table 2. In the two liquid environments, JP-4 jet fuel and JP-X rocket fuel, the wear characteristics of a selected number of materials rubbing against washer specimens of hardened AISI 440 C stainless steel were evaluated. This steel was selected as a common wear component in the liquid environments, since it is frequently used for parts in APU and rocket motor mechanisms that require a hardenable, corrosion-resistant material.

Of the prospective seal materials that were studied in the JP-4, a bonded boron nitride, a synthetic mica, and two mica-containing cermets were found to have friction and wear properties at least as favorable as a conventional graphite. The best of these, a boron nitride body and a nickel-mica cermet, worked so well that the wear tracks were hardly visible on the specimens after their evaluation. An experiment with a representative, wear-resistant alloy revealed an extraordinarily low coefficient of friction for this material rubbing against the stainless steel in JP-4. However, as shown in Figure 3, the surface damage on these specimens would seem prohibitive in a seal application. It is possible that the unusually smooth surface finish on the specimens in this evaluation might have accounted for their low friction during normal operation. The surface damage could have occurred during the deceleration of the rotating specimen at the end of this experiment. The alumina porcelain and Alumina Coating A were the only specimens to fail before their tests were completed. A photograph of the specimens after the experiment with the alumina porcelain is shown in Figure 4. From the standpoint of friction and wear, this was the poorest combination evaluated in the liquid environments.

Two of the materials that were investigated in the JP-4 environment were re-evaluated in JP-X. Neither one showed exceptional promise as a seal material in this new rocket fuel. The boron nitride material suffered considerable surface damage leaving deposits on the stainless steel, even though only a slight amount of wear could be measured. The graphite specimens incurred more surface damage and wear in the JP-X in 45 minutes of total operating time than they incurred in the JP-4 in 95 minutes. In fact, the JP-X caused the graphite vanes in the supply pump to wear so rapidly that further experiments in this liquid with the present pump were prevented. In the two evaluations that were made, the flow rate of the rocket fuel was somewhat erratic, possibly influencing the wear of the specimen materials.

Of the evaluations conducted in the hot oxidizing gaseous environments, only one set of specimens operated without surface failure long enough for the temperature to be increased to 1400 F. This set, boron carbide buttons rubbing against alumina porcelain, operated with friction coefficients between 0.31 to 0.47 at gas temperatures up to 1200 F. However, when the environmental temperature was increased to 1400 F, the friction rose to 0.55. After about one minute of operation under a load of 5-psi contact pressure in 1400 F air, the coefficient of friction jumped suddenly to 0.94, indicating a very high rate of heat generation at the rubbing surfaces. About 30 seconds after this sudden increase in friction both the rotating specimen and the button specimens shattered, causing the immediate shutdown of the equipment. Possibly

TABLE 2. RUBBING WEAR EVALUATIONS OF PROSPECTIVE

Evaluating Environment	Washer Specimen Material	Button Specimen Material	Wear Surface Roughness Before Evaluation, microinches, rms	
			Washer	Button
200 F, JP-4, jet fuel	AISI 440 C stainless steel, hardened to 56 R_C	Graphite A	0.5	7-12
Ditto	Ditto	Alumina porcelain	0.2	8-11
"	"	Boron nitride	0.6	25-35
"	"	Nickel-boron nitride-mica	0.7	15-25
"	"	Cobalt-base Alloy A	0.7	8.1
"	"	Alumina Coating A on stainless steel	0.5	15-20
"	"	Synthetic mica	0.6	4-6
"	"	Nickel-mica	0.7	5-6
800 F, JP-X, fuel	"	Graphite A	0.6	7-12
Ditto	"	Boron nitride	0.3	15-20
Oxidizing (essentially air), up to 800 F(f,h)	AISI 440 C stainless steel, process annealed	Graphite B	1.5-2	--

BEARING AND SEAL MATERIALS[a]

Dynamic Behavior at 20 Psi Contact Pressure			Observations of Specimens After Evaluation	
Friction Coefficient	Max. Specimen Temp, F	Total Operating Time, minutes	Washer	Bottom
0.05	193	95	No wear except a slight polishing	Slight scoring, no measurable wear
0.11 - 0.16[b]	203	85	Scored, 10 mils wear, surface roughness increase of 46 microinches, rms	Crushed and scored, 8 mils wear, surface roughness increase of 18 microinches, rms
0.03	178	96	No wear except a slight polish on the edges of the wear track	No wear, lapping scratches still visible
0.04	190	105	No wear except a slight scar mark in the middle of the wear track	Some scoring 0.1 mil wear
0.02	188	95	Scored, 0.32-mil wear, surface roughness increase of 15 microinch, rms	Wear depends on surface, surface roughness increase of 2-12 microinches, rms
0.05[b,c]	185	51	Scored, 0.34 surface roughness increase of 25-30 microinches, rms	Scored, 0.61 mil wear, surface roughness increase of 25-40 microinches, rms
0.04	190	115	No wear except a slight polishing, no roughness increase	Slight wear, 0.02 mil, surface roughness increase of 3-9 microinch, rms
0.03	194	113	No wear except a slight polishing	No wear except a slight polishing
0.04[b,d]	50	45	No wear, some deposits on the wear track	Some scoring, 0.5 mil wear
0.04[b,d,f]	80	39	Wear deposits on surface, surface roughness increase of 4 microinches, rms	Badly scored, 0.1 mil wear
0.25[b,c]	[illegible]	38	Wear deposits on surface, surface roughness increase of 5 microinches, rms	Badly scored, 1.4 mils wear

TABLE 2.

Evaluating Environment	Washer Specimen Material	Button Specimen Material	Wear Surface Roughness Before Evaluation, microinches, rms	
			Washer	Button
Oxidizing (essentially air), up to 400 F	Alumina coating 8 on stainless steel	Nickel-mica	0.6	15
Oxidizing (essentially air), up to 1400 F	Alumina porcelain	Boron carbide	13	--

(a) In each experiment three button specimens were held against a rotating washer specimen, so that the contacting surfaces were parallel to each other. The rubbing speed was 200 feet per second.

(b) Failure was indicated by a sharp rise in the friction and noise level before the specimens could be operated for one hour at 20-psi contact pressure.

(c) The maximum load pressure in this evaluation was 15 psi.

(d) A diminishing amount of rocket fuel was supplied to the rubbing surfaces during these evaluations, since this fluid caused excessive wear on the graphite vanes in the supply pump.

(Continued)

Dynamic Behavior at 20 Psi Contact Pressure				
Friction Coefficient	Max. Specimen Temp, F	Total Operating Time, Minutes	Observations of Specimens After Evaluation	
			Inside	Bottom
0.12[b,c]	445	8	Sound, wear depths on surface, surface roughness increase of 20-30 microinches, max	Body sound 2.7 mils wear
0.55[b,c]	1345	56	Shattered during failure	Shattered during failure

(c) The maximum load pressure in these evaluations was 5 psi.
(f) The specimens in these evaluations failed before the air temperature could be raised to 1460 F.
(g) The maximum load pressure in this evaluation was 10 psi.
(h) Alignment difficulties in the apparatus limited the temperature in this evaluation.

Note: The composition and selected properties of the materials are found in Table I.

FIGURE 3. BUTTON SPECIMENS OF COBALT-BASE ALLOY A, AND THE WASHER SPECIMEN OF HARDENED STAINLESS STEEL, AFTER RUBBING WEAR EVALUATION IN JP-4 JET FUEL

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FIGURE 4. BUTTON SPECIMENS OF ALUMINA PORCELAIN, AND THE VARIOUS SPECIMEN OR HARDNESS IMPRESSIONS, AFTER TESTING WEAR EVALUATION IN UP-4 TEST FUEL

the thermal shock from the frictional heat generation imposed excessive stresses in the ceramic specimens.

In the other two rubbing wear evaluations in the hot gaseous environment, the specimens also exhibited premature surface failures, although they were at lower temperatures and were not as violent as in the evaluation just described. Even though the temperature of the environment in the experiment with the oxidation-resistant graphite rubbing against AISI 440 C stainless steel was limited to 800°F, the wear and surface damage that occurred indicates that this combination would not be desirable in a seal. In the evaluation of the nickel-cermet rubbing against the Alumina Coating B, the coefficient of friction increased suddenly from 0.18 to 0.57 after only 90 seconds of operation at a load pressure of 5 psi. Several minutes of continued operation, with the friction oscillating erratically from 0.27 to 0.57, produced the surface damage shown in the photograph in Figure 9.

Static Corrosion Studies

A liquid-rocket-motor propellant that holds considerable promise is the oxidizer, inhibited red fuming nitric acid (IRFNA). From a list of possible candidate materials for seal and bearing applications in this liquid, thirteen were selected and given a screening-type evaluation in IRFNA to determine which materials should be eliminated from further consideration due to excessive attack in this strongly corrosive medium. Only those materials which were thought to have some possibility of withstanding the attack of IRFNA and those which represented a general class of commercially available ceramics, cermets, or alloys were selected for these screening studies.

These evaluations paralleled those which have been previously conducted and reported by [illegible] Laboratory at Wright Field (WADC TR 55-337, Robinson and Mather). In this study, the corrosion specimens were exposed to IRFNA in an autoclave for six days at 160-175 F.

Approximately 1.5 liters of IRFNA was supplied in an aluminum container by WADC for these experiments. Hydrofluoric acid is the inhibitor used in this acid, and it is added to inhibit the corrosion of the stainless steel and aluminum containers that are used for storing red fuming nitric acid (See WADC TR 59-109, Phelps, Lee, and Robinson). The analysis of the acid which was supplied is compared with the Military Specifications for Type III-A IRFNA in Table 3.

TABLE 3. COMPOSITION OF IRFNA

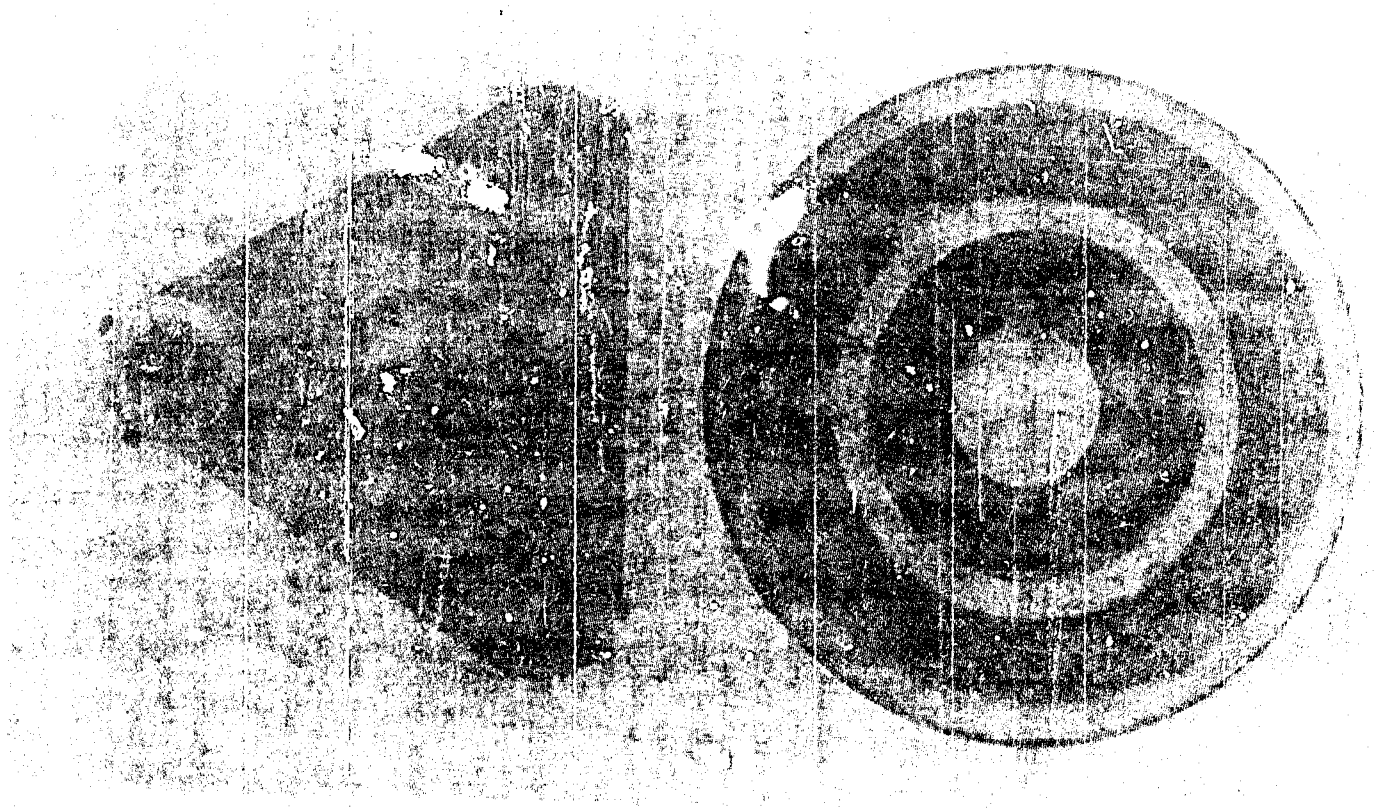
Component	Stock Acid, per cent	MIL-N — 7254B (USAF), per cent
HNO_3	86.8	81.3 - 84.5
NO_3	9.9	14 ± 1.0
H_2O	3.2	2.5 ± 0.5
Ash	0.03	0.1 max
HF	0.6	0.6 ± 0.1

Description of Apparatus Used in Static Corrosion Studies

Teflon-lined Monel autoclaves were used to contain the IRFNA during the exposure period. These were small stainless bombs holding about 55 ml when assembled. Two of these were fitted with Teflon stoppers through which extended a flared length of stainless steel pressure tubing. By tightening a suitable arrangement of fittings after the cap had been threaded onto the bomb, the flared end of the pressure tubing could be drawn into the Teflon plug, thus expanding it against the Teflon-lined wall of the bomb. This formed a simple Bridgeman-type closure which tends to seal better as more pressure is exerted within the bomb. A two-way stainless steel pressure valve was attached to the end of the pressure tubing. One outlet from this valve was used for filling and bleeding off excess pressure from the bomb. The other was connected to a 0 to 100-psi stainless steel pressure gage. The pressure tubing connecting the gage to the valve was bent into a loop. This loop, when partially filled with Kel F 10, a fluorolube oil, served as a trap to prevent the highly corrosive vapors from reaching the mechanism of the gage.

This assembly was exposed to IRFNA at 160 F with no specimens present. Subsequent analysis of the acid for copper and nickel showed, by the absence of both of these elements, that no attack of the Monel walls of the autoclave had occurred by acid leaching through the Teflon liner.

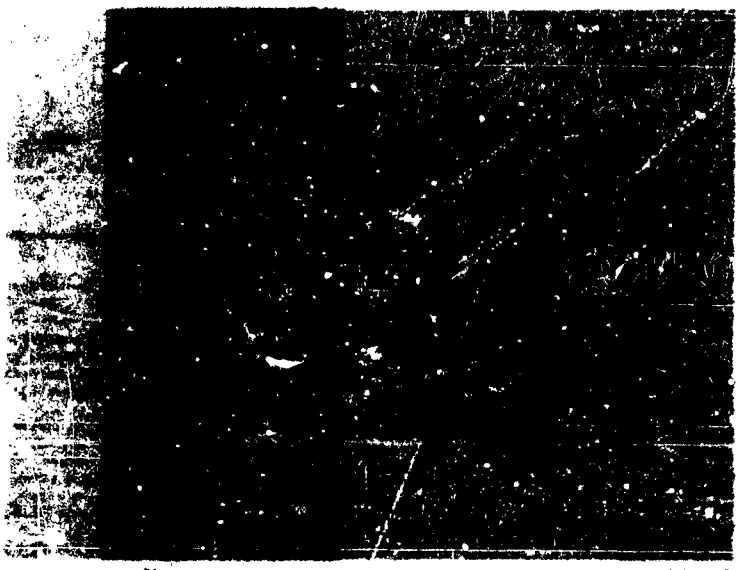
The corrosion specimens were machined into round buttons, 1/2 inch in diameter by 1/4 inch long. The one exception was the aluminum-chromium specimen which was irregularly-shaped but of approximately equivalent dimensions. One face of each specimen was lapped to a high finish to facilitate the examination of the corrosive surface damage. Prior to exposure, all specimens were cleaned with 10-kc sonic vibrations, using detergent and water, and then oven dried and weighed. Special holders cut from Teflon sheet were used to hold the specimens submerged in the acid



N35812

FIGURE 5. BUTTON SPECIMENS OF NICKEL-MICA, AND THE WASHER SPECIMEN OF ALUMINA COATING B ON STAINLESS STEEL, AFTER RUBBING WEAR EVALUATION IN A 600 F OXIDIZING ATMOSPHERE.

inside the bomb. This is illustrated in Figure 6 which shows the specimens, holder, and exploded view of the bomb and closure device.



Approx. 1/2X M35736

FIGURE 6. AUTOCLAVE WITH CLOSURE, SPECIMEN HOLDER, AND SPECIMENS FOR THE STATIC CORROSION STUDIES IN IRFNA

Test Procedure and Results of Static Corrosion Studies

The procedure that was followed consisted of placing four or five cleaned and weighed specimens in a holder inside the bomb. The bomb was then sealed and placed in an electrically heated water bath. After all the tubing connections had been made, the system was evacuated and allowed to stand until it was certain that a good seal had been obtained. Once this had been established, 40 ml of IRFNA was added by means of suction using a polyethylene funnel and tube. The system was vented to assure a starting pressure of one atmosphere, and then it was closed. The bath was heated to 160 F and held at this temperature for six days. If four specimens were used, the ratio of acid volume to surface area of specimens was about 12.7 ml/sq in.; five specimens gave a ratio of about 10.1 ml/sq in. There was about 26 per cent ullage in each bomb. The pressure which built up in the bombs during exposure was an indication of the amount of corrosion taking place. In the absence of specimens, a pressure of 26 psi was reached. For the reasonable amount of corrosion that occurred in the first two bombs, the pressure reached 40 to 50 psi and in the bomb containing the last five specimens, where corrosion was decidedly severe, the pressure reached 100 psi after 3 days' exposure. This bomb was bled until the pressure was 35 psi, but it climbed to 62 psi before the end of the test.

TABLE 4. RESULTS OF THE STATIC CORROSION TESTS OF PROSPECTIVE SEAL AND BEARING MATERIALS EXPOSED TO IRFNA

(Six Days' Exposure, 1440°)

Material	After Exposure: Weight Loss, mg	After Exposure: Weight Change, per cent	After Cleaning: Weight Loss, mg	After Cleaning: Weight Change, per cent	Observations of Specimens After Cleaning
WC-TaC-Co	489.7	-3.84	460.2	-3.60	Gray coating, etched
WC-platinum	136.2	-1.06	136.1	-1.07	Gray coating, etched
Boron carbide	+1.2	+0.06	0.0	0.00	Slight film removed by cleaning
Chromium carbide-nickel	13.0	-0.21	16.3	-0.27	Slightly etched
Synthetic mica	1.9	-0.09	6.1	-0.27	Slightly etched
Nickel-mica	26.9	-0.69	48.2	-1.20	Etched, selective attack of Ni
High-density alumina	0.1	-0.003	0.5	-0.02	Unchanged from original
Alumina-chromium	4.6	-0.08	5.9	-0.11	Slightly etched
Zirconia coating on AISI 440 C stainless steel	2629.2	-42.49	2603.6	-40.56	Large surface pits
AISI 440 C stainless steel	2993.3	-46.28	2088.9	-40.36	Small surface pits
Cobalt-base Alloy A	40.1	-0.57	43.3	-0.61	Surface eroded and cracked
Cobalt-base Alloy B	18.4	-0.27	19.0	-0.28	Slightly etched
Iron-base Alloy C	1930.0	-26.11	1900.0	-25.76	Surface eroded

Note The iron-base alloys, AISI 440 C stainless steel and Iron-base Alloy C, were excessively attacked. This attack occurred in a uniform manner so that the specimens were decreased uniformly in dimensions without losing their original shape.

The average area per specimen was about 8.8 sq in.

The composition and selected properties of the materials are listed in Table 1.

At the end of the exposure period, the bombs were cooled, vented of excess pressure, and opened. The specimens were thoroughly rinsed with water and oven dried. The specimens were weighed, and cleaned by 10-kc sonic vibrations in detergent and water to loosen any particles or corrosion products which could be removed by a scrubbing action; then, they were reweighed.

The results of these screening-type evaluations are given in Table 4. Since the experiments were designed only to select the materials which are practically unattacked by IRFNA, the corrosion is expressed in percentage of change in weight from the original weight. From these results, it would appear that boron carbide and high-density alumina show the most promise as materials for seal and bearing application in IRFNA from a corrosion standpoint. It is possible that the surface heating and resultant stresses arising from use of these materials as bearings and seals might alter their corrosion resistance to a considerable extent. For this reason, it must be remembered that these are screening-type evaluations and must be followed by tests in which the material is serving its intended use in IRFNA before a precise picture of the applicability of these materials can be obtained.

Since some missile applications do not call for protracted exposure of the rocket motor to the propellants, it might be well to consider for further evaluation some of the other materials which were only slightly attacked during the six-day exposure. Such materials as alumina-chromium, synthetic mica, chromium carbide-nickel, and the cobalt-base alloys might be considered, in the order given, in this category.

DISCUSSION AND CONCLUSIONS

The preliminary results that were obtained in this program in the high-speed-rubbing wear apparatus corroborates somewhat the materials-compatibility information from field service experience that was learned in the aforementioned survey. The satisfactory behavior of mica-containing materials in some liquid environments and the abrasion and excessive wear of alumina porcelain with itself and with stainless steel in some applications was substantiated by the experimental results in the JP-4 environment. Also, the superiority of the boron carbide versus alumina porcelain combination in the hot gas might be expected in the light of some field experience. It would seem, therefore, that the equipment and procedures which were developed and used in this study provided a realistic evaluation of materials for seals and bearings in aircraft accessory equipment and rocket motor applications.

Several materials performed satisfactorily in the rubbing wear experiments that were conducted in JP-4 jet fuel. Since this environment did not appear to present any processing material problems in these evaluations, it is possible that materials are available for most future, as well as present, bearing and seal applications in this fluid.

However, the experimental results indicate that the future use of JP-X rocket fuel and nitric acid oxidizer (IRFNA) in missile systems might intensify the materials problems in seal and bearing components that are exposed to these two environments.

The graphite and boron nitride materials that were successful in the JP-4 evaluations suffered an appreciable amount of surface damage and wear in the JP-X. Evidently, the JP-X affected these materials in such a way as to seriously hamper their lubricating mechanisms. Practically all of the materials that were evaluated for static corrosion resistance in the nitric acid were attacked to some extent in this strongly corrosive environment. Gross corrosion of a material would make it unsatisfactory for bearing or seal applications from a structural viewpoint. Even slight indications of corrosive attack are important in the evaluation of rubbing wear materials since the character of the surface determines its friction and wear properties. Wear and friction may increase as a result of surface roughening by the corrosive action. Also, rubbing wear can accelerate corrosion by removing the corrosion products and presenting a fresh surface to the environment. On the other hand, the slight amount of corrosion in some of these static experiments may indicate the presence of a surface reaction that hopefully would contribute to the lubrication of the material in a manner similar to that in which oxide films prevent the surface welding and scoring of some metals (W. Hirst and J. K. Lancaster, "The Influence of Oxide and Lubricant Films on the Friction and Surface Damage of Metals", Proceedings of the Royal Society of London, Vol 223, May 6, 1954, p 324).

The data from the experiments that were conducted in the hot oxidizing gaseous environment indicate the severity of this atmosphere on the rubbing wear performance of the materials evaluated. Apparently the combined corrosion and heat effects were sufficient to disrupt any surface lubrication mechanisms that might have prevented excessive damage and wear. The incorporation of rubbing seals and bearings in hot oxidizing gases will probably be delayed by the lack of suitable materials.

It appears that in the three unusual environments, JP-X, nitric acid, and hot oxidizing gas, the usual mechanisms of lubrication and wear resistance in seal and bearing materials might be adversely affected by the corrosive agents present. A more thorough understanding of rubbing contact lubrication, i.e., the mechanism by which material surfaces are kept separated enough during rubbing contact to prevent excessive wear and abrasion, is needed if materials are to be developed for the variety of atmospheres that are expected in future aircraft power plant systems. It is possible that entirely new mechanisms of surface film generation and maintenance will have to be developed for each of these environments. Hopefully, prospective seal and bearing materials might be designed to operate in these fluids under the protection of lubricating surface phenomena similar, perhaps, to those found in extreme pressure films and layer-lattice type structure materials (like graphite) in conventional applications.

RECOMMENDED FUTURE WORK

The development and fabrication of new materials for elevated temperature and corrosive applications has increased tremendously in recent years. However, very little effort has been devoted toward the tailoring of such materials for rubbing wear applications such as are found in bearings and seals. The mechanisms of wear,

lubrication, and surface friction in the extraordinary environments surrounding some APU and rocket bearings and seals can be quite different from those ordinarily encountered. Such environmental fluids might well disrupt the regenerative mechanism of surface films, found to be so important in the lubrication of some materials having a layer-lattice-type structure (V. R. Johnson, "Investigation of the Mechanism of Molybdenum Disulfide Lubrication in Vacuum", Journal Applied Physics, Vol 27, October, 1956, p 1175). Materials of this type of structure have been successful for many years in seal and bearing applications (graphite), and effort should be devoted toward the adaptation of similar mechanisms of lubrication to elevated temperature and chemically corrosive environments.

It is recommended, therefore, that an extensive program of materials evaluation and development be initiated toward the solution of these unusual lubrication problems. The APU and rocket motor manufacturers need materials for bearing and seal components in equipment that is now being developed. The wear applications in anticipated future power plants are expected to be even more demanding of materials. The technological advancement of lubricants and materials for rubbing wear components has lagged behind the general aircraft power plant improvements of recent years, and it threatens to seriously hamper advanced design plans for future aircraft.

The development of the experimental apparatus and the evaluation procedure for the comparison of prospective seal and bearing materials under conditions of high-speed rubbing wear has been accomplished in this program. A comparison of the experimental results with known field experience has indicated that the lubrication and wear conditions in a face-type shaft seal have been simulated in this equipment, which utilizes small specimens of simple shape. Thus, the wear characteristics of materials of which it would be difficult to fabricate into full size seal rings can be evaluated quickly and economically. The study of the lubrication and frictional phenomena of experimental materials under high-speed rubbing wear in elevated temperature gaseous environments can be easily accomplished with this apparatus. The rubbing wear evaluation of materials in the more corrosive liquid environments (JP-X, IRFNA, etc.) can be conducted in a similar apparatus that is suitably adapted for handling these liquids. Since a program for the study of prospective bearing and seal materials for operation under the environmental conditions encountered in present and future aircraft accessory equipment and rocket motor applications appears to be both feasible and profitable, the initiation of such a program is highly recommended.

(Data for this report are recorded in Battelle Laboratory Record Books No. 9400, pages 30 through 59 and pages 62 through 68, and No. 11342, pages 4 through 11, inclusive.)